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**TWELFTH TECHNICAL REPORT**

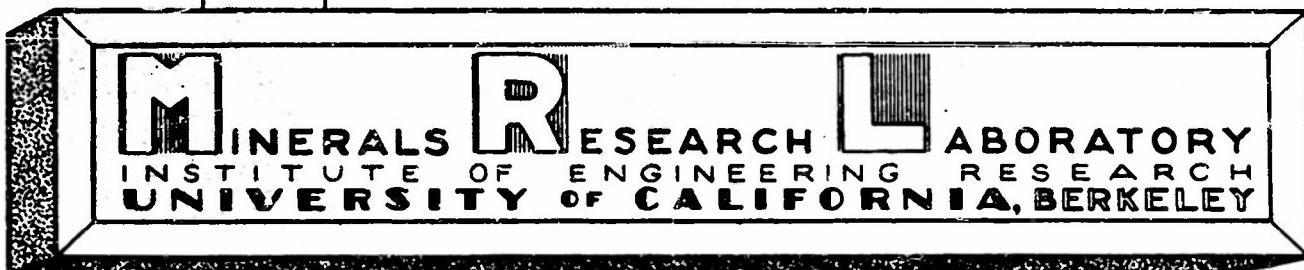
**LOW TEMPERATURE CREEP  
OF ZINC SINGLE CRYSTALS**

**BY**

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Twelfth Technical Report

Low Temperature Creep of Zinc Single Crystals

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## ABSTRACT

Creep of zinc in the temperature range from -45° C to 0° C was investigated by means of spherically shaped single crystal shear specimens. It was found that creep curves could only be reproduced when the following conditions were met: (1) the annealing temperature was near the melting point of zinc, and (2) the loading time was kept constant.

The initial part of the creep curves could be represented by the equation  $E - E_0 = A \log t$  where  $E$  is the creep strain,  $E_0$  is the instantaneous strain,  $A$  is a constant and  $t$  is the time. However, the latter part of the plot usually curved upward. The time at which the creep curve deviated from the logarithmic creep, defined as the transition time, was found to be temperature dependent. The activation energy associated with the transition time was about 19,000 cal/mole; it was independent of the applied stress. This suggested that diffusion was the rate controlling factor governing the transition time.

The experimental results of this investigation also suggested that the controlling factor of logarithmic creep seemed to be the back-stress rather than the thermal activation stress, as proposed by the exhaustion theory.

## Introduction

Experimental evidence has shown that slip is the major process of creep (1-4). At high temperatures dislocations can climb from one slip plane to another whenever imperfections are encountered (5,6). At low temperatures, on the other hand, the mobility of the dislocations is limited because they cannot climb over the imperfections which cause dislocations to pile up. The phenomenon of creep is, however, not confined to high temperatures; this fact has been known since 1905 (7,8). Creep has been observed in cadmium and zinc single crystals at a temperature as low as 1.5°K (9). The difference between high temperature and low temperature creep may be viewed as a difference in the controlling factor—in the case where sufficient thermal energy is available, the controlling factor is diffusion, whereas low temperature creep is controlled by the distribution of imperfections and the stress fields associated with them.

Unfortunately, few extensive investigations of low temperature creep behavior have been made. Only after Orowan (10) proposed his structural hardening theory and Mott and Nabarro (11) and Smith (12) their exhaustion theory did it become recognized that low temperature creep might help provide an understanding of the mechanism of metal plasticity.

One characteristic of low temperature creep is the extremely small magnitude of creep strain, as compared with the instantaneous strain. For example, Wyatt (13) observed that the creep strain of a copper specimen at liquid nitrogen temperature under a constant stress of 35,200 psi for a period of 1,000 seconds was only 0.002 in/in whereas the instantaneous strain

was 0.060 in/in. In other words, the creep rate diminishes so rapidly that the instantaneous strain is practically all that can be seen. Because of the small magnitude of strain involved, low temperature creep may well be suspected to be due to processes other than slip, e.g., subgrain formation (14), grain boundary shearing (4), etc. In order to remove the confusion which exists and simplify theoretical consideration, it would be desirable to use specimens subjected to simple slip. Simple shear single crystal specimens, whose advantages have been fully discussed by Parker and Washburn (15), seem to satisfy this condition.

The present investigation is a study of low temperature creep by means of simple shear single crystal specimens. The purpose of the investigation was to determine, if possible, the nature of low temperature creep.

### Experimental Technique

Spherical single crystals of 99.99% pure zinc one inch in diameter were grown in a graphite mold in an electric furnace in a helium atmosphere. The furnace was built in such a manner that it had a temperature gradient of approximately  $5^{\circ}\text{C}$  per inch, being hotter at the top and cooler at the bottom. The furnace was heated until the temperature at the bottom of the mold was  $100^{\circ}\text{C}$  above the melting point of zinc; then the power input was turned down so that the equilibrium temperature of the furnace was about  $100^{\circ}\text{C}$  below the melting point. The furnace, because of its high heat capacity, required twenty hours to cool to the final temperature.

For determining orientation, the crystals were polished in concentrated nitric acid and then etched in 50% hydrochloric acid until the hexagonal etch pattern was clearly revealed. The crystals were next cooled to liquid nitrogen temperature and cleaved along the basal plane (0001) with a sharp knife edge. The slip direction [ $\bar{2}110$ ] was determined by cleaving a thin layer of the basal plane half way across the crystal; the line of fracture followed the [ $\bar{2}110$ ] direction (the slip direction).

Next, the crystals were acid-machined into a dumb-bell shape for simple shear tests. The test section was cylindrical in shape, having a length of 0.125 inch and a diameter of 0.653 inch. The gripping assembly for simple shear tests is shown in Fig. 1. The crystallographic alignment of the specimen with respect to the external load was such that the [ $\bar{2}110$ ] direction was parallel to the shearing force.

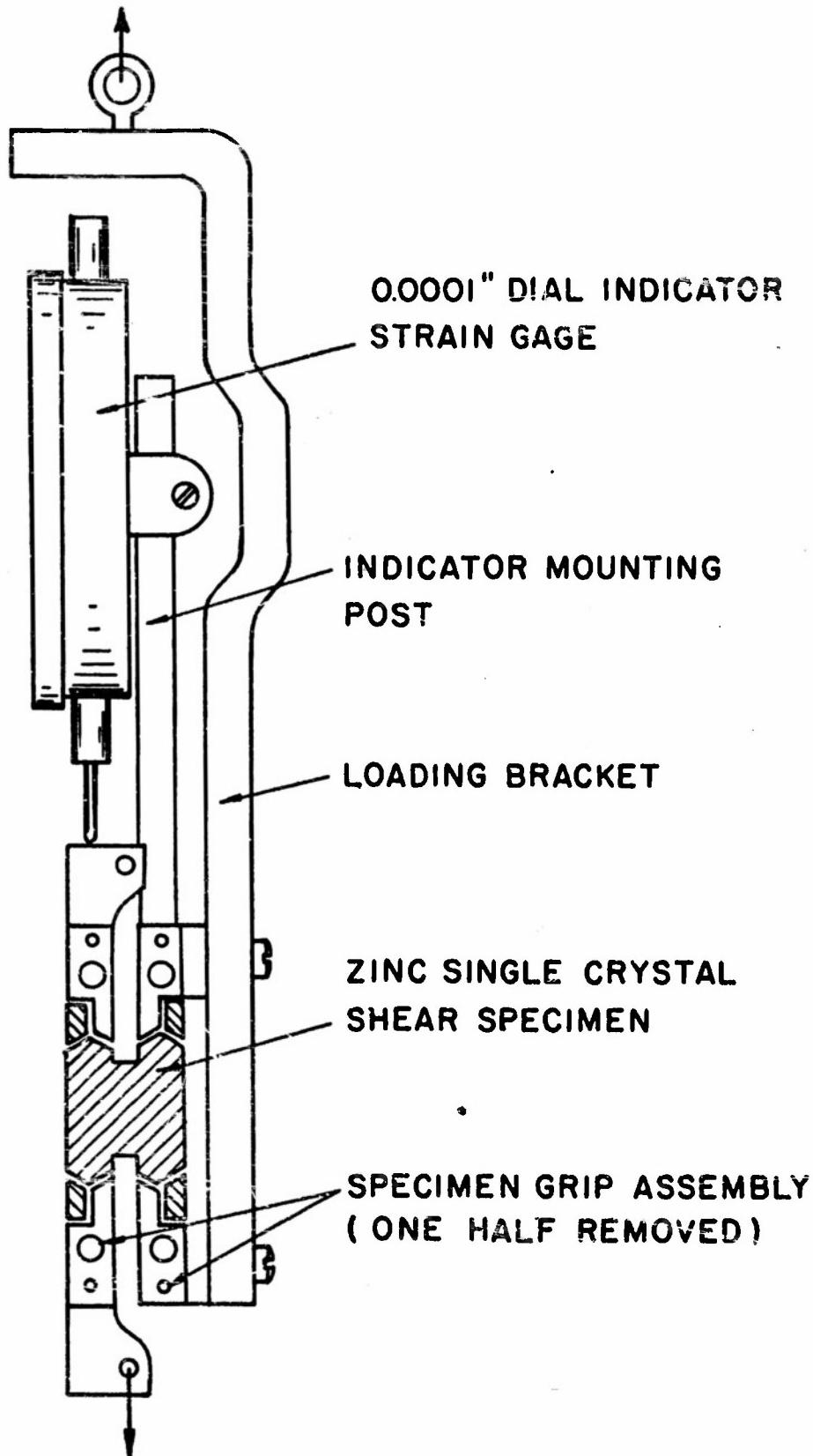


FIG. I SKETCH OF LOADING AND STRAIN  
MEASURING ASSEMBLY

The creep testing machine was of the conventional level-arm type. However, the level arm ratio was only 1:1.25, since the maximum load in this investigation never exceeded 15 pounds. A set of dead weights hung on the longer arm of the machine provided the necessary load. The loading time was adjusted so that it was fast enough to establish the initial rate of creep but was slow enough to avoid impact conditions. This was accomplished by means of a hydraulic jack with a constant-speed moving head which applied the weight to the specimen.

The actual load on the specimen was measured directly with a dynamometer mounted on the extension rod of the specimen. The dynamometer also served two additional purposes, (1) to determine the shortest time for loading without perceptible impact, and (2) to mark the time at which instantaneous strain ends and creep begins. The strain was calculated from the readings of an 0.0001" dial gauge extensometer. Usually, toward the latter part of the test the creep strain would become so small that for accurate reading of the extensometer a low power microscope had to be used. With 50 magnification, extensions up to  $2 \times 10^{-6}$  inches could be observed; this corresponded to a strain of  $1.6 \times 10^{-5}$ . Fig. 2 shows the creep test set up schematically.

For tests at  $0^{\circ}\text{C}$ , the specimen was immersed in a Dewar flask containing water-ice. For tests at the temperature range between  $-50^{\circ}\text{C}$  and  $-10^{\circ}\text{C}$ , the specimen was immersed in a Dewar flask containing ethyl alcohol. The temperature of this bath was maintained constant in the manner shown schematically in Fig. 3. The ethyl alcohol was circulated by an electrical pump

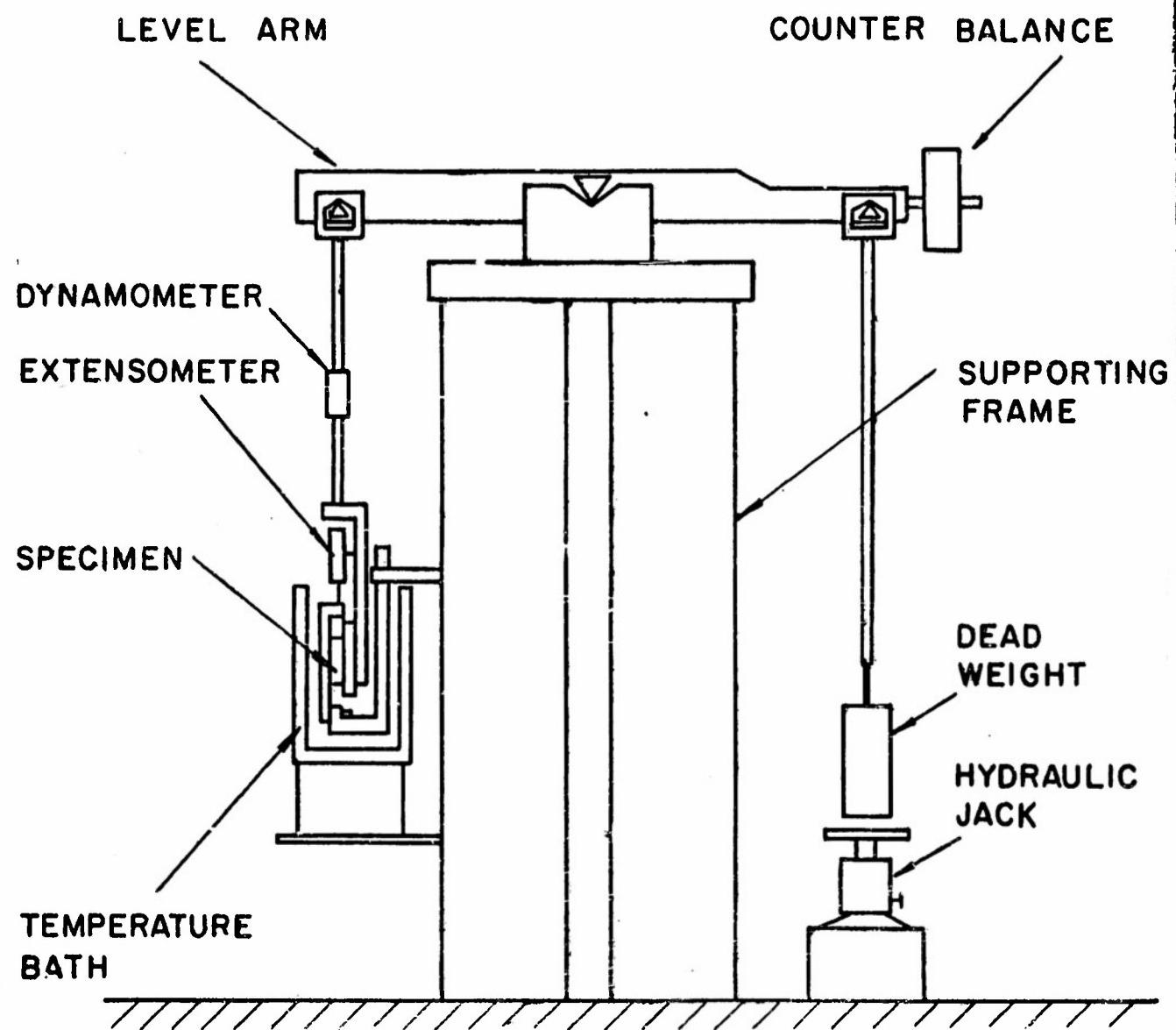


FIG. 2 SCHEMATIC DIAGRAM OF THE LOADING MECHANISM FOR CREEP STUDIES .

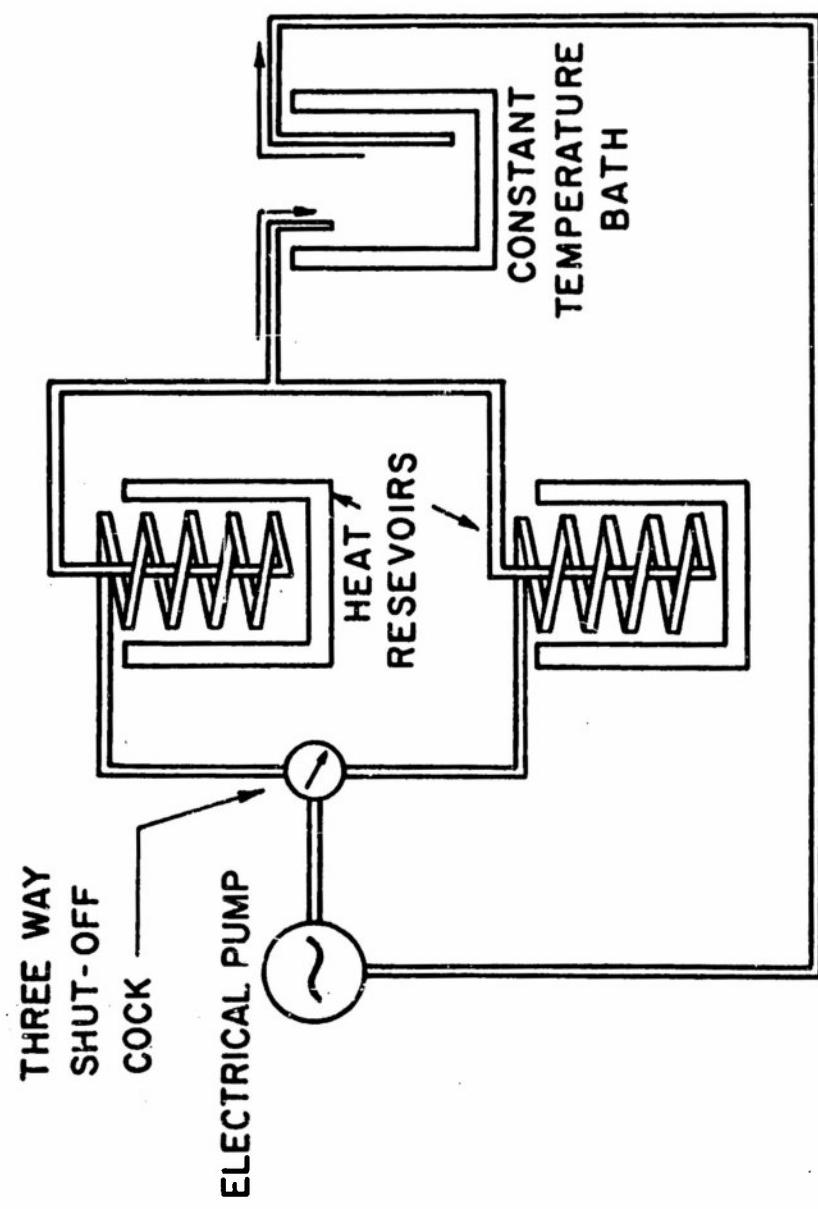


FIG. 3 SCHEMATIC DIAGRAM OF THE CIRCULATION SYSTEM USED FOR CONSTANT TEMPERATURE CREEP STUDIES

through aluminum pipes. These pipes were immersed in two constant-temperature heat reservoirs, one containing ethyl alcohol and dry ice, the other water and ice. The temperature of the ethyl alcohol circulating in the pipes was controlled by a 3-way shut off cock which could be used to adjust the amount of alcohol going through each of these two heat reservoirs. The testing temperature could thus be maintained constant within 1°C.

### Experimental Procedure and Results

Reproducibility of experimental results is a necessity. A previous investigation(16) had revealed that annealing at 400°C for one hour could completely remove all the previous cold working so that stress-strain curves could be reproduced. This reproducibility was confirmed by Edwards and Washburn (17). A careful review of the work by Brown, Washburn and Parker (18) on room temperature creep with the same kind of specimens showed that the annealing treatment they employed (260°C for one hour) was not close enough to the melting temperature of zinc to permit reproduction of the creep curve.

In the present investigation, the annealing procedure was standardized as one hour at 400°C followed by furnace cooling to room temperature; the total cooling time was  $2\frac{1}{2}$  hours. To avoid oxidation of the specimen surface, annealing was carried out in a helium atmosphere. The loading time for creep tests was standardized at 4 seconds. This particular time was found to be the shortest possible loading time without introducing perceptible impact. With these precautions reproducibility of the creep curve was achieved.

Fig. 4 shows the reproducibility of creep curves from a same single crystal. It should be noted that although the creep curves were reasonably well reproduced, the instantaneous strain was not consistent. For example, for the tests at -7.5°C, the instantaneous strain varied from 0.0364 to 0.0382. On the other hand, the instantaneous strain was not as sensitive

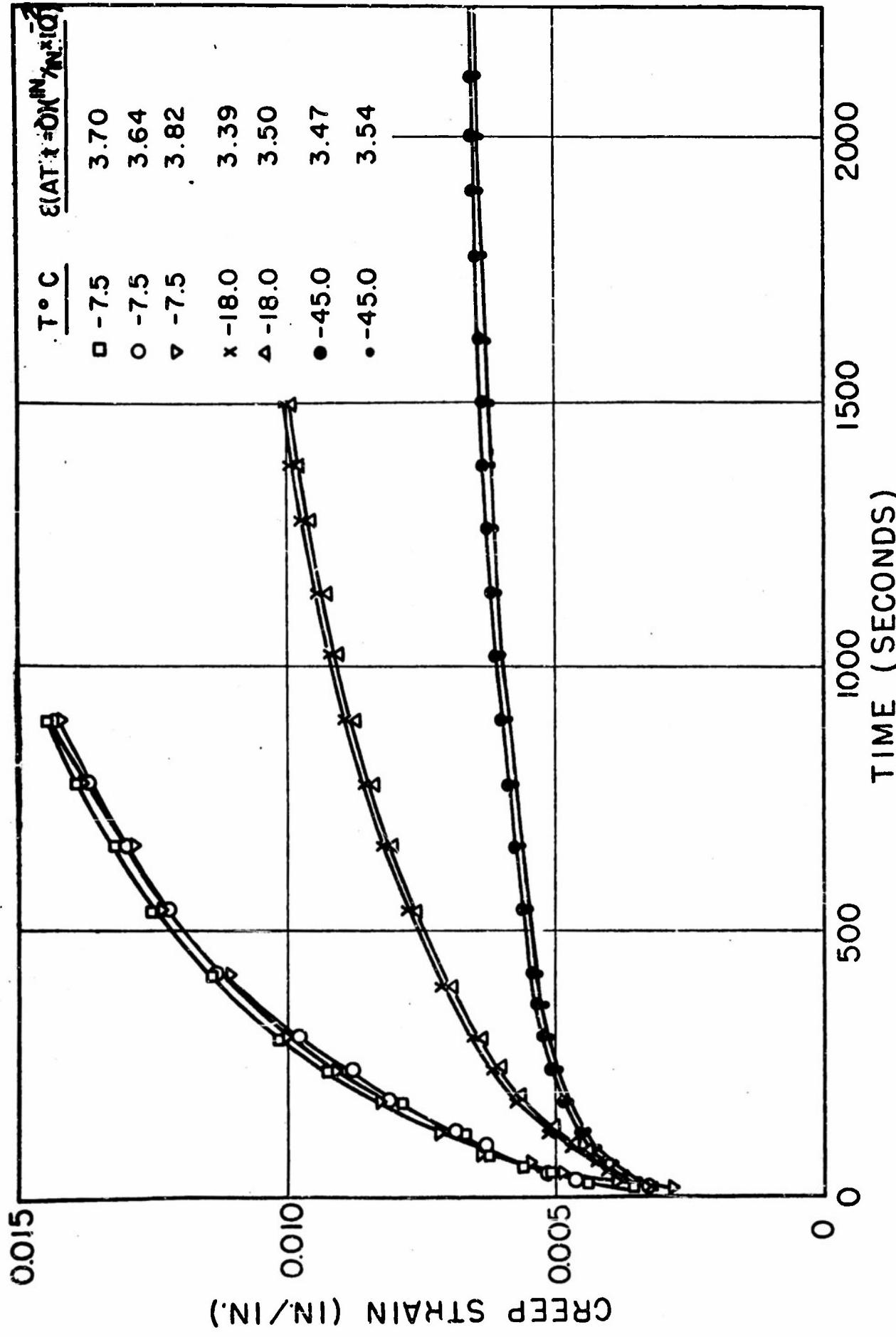


FIG. 4 CREEP CURVES OF A Zn SINGLE CRYSTAL UNDER A CONSTANT SHEAR STRESS OF 34.1 PSI AT THREE DIFFERENT TEMPERATURES.

to temperature as was the creep strain. As demonstrated by the creep curve in Fig.4, under the same constant shear stress of 34.1 psi the creep strain for 900 seconds was 0.0145 at  $-7.5^{\circ}\text{C}$ , almost two and a half times as that at  $-45^{\circ}\text{C}$ ; but the instantaneous strain at  $-7.5^{\circ}\text{C}$  was only 8% larger than that at  $-45^{\circ}\text{C}$ .

With regard to the creep curves themselves, Fig.4 shows that at  $-45^{\circ}\text{C}$  the rate of creep is high in the first few minutes but diminishes rapidly with time. For example, at 10 seconds, the creep rate was  $1.7 \times 10^{-4}$  per second, and at 2,000 seconds,  $8.5 \times 10^{-7}$  per second. Philips (7) suggested that the low temperature creep curves could be represented by the following equation:

$$\mathcal{E} - \mathcal{E}_0 = A \log t$$

where  $\mathcal{E}$  = creep strain

$\mathcal{E}_0$  = instantaneous strain

$A$  = constant

$t$  = time

A replotting of data from Fig. 4 on a semi-log scale as shown in Fig.5, demonstrated that the curves obtained at  $-45^{\circ}\text{C}$  followed this behavior up to the end of the test. Those obtained at the other two temperatures ( $-7.5^{\circ}\text{C}$  and  $-18^{\circ}\text{C}$ ) were straight lines for the initial portions only and curved gradually upward. These observations seem to be in line with observations made by Davis and Thompson (19) Wyatt (13), Philips (7), Chevenard (20), Laurent and Eudier (21) and Haasen and Leibfried(22).

It is interesting to note that the straight line portions in this semi-log plot are parallel in the temperature range between  $-7.5^{\circ}\text{C}$  and  $-45^{\circ}\text{C}$

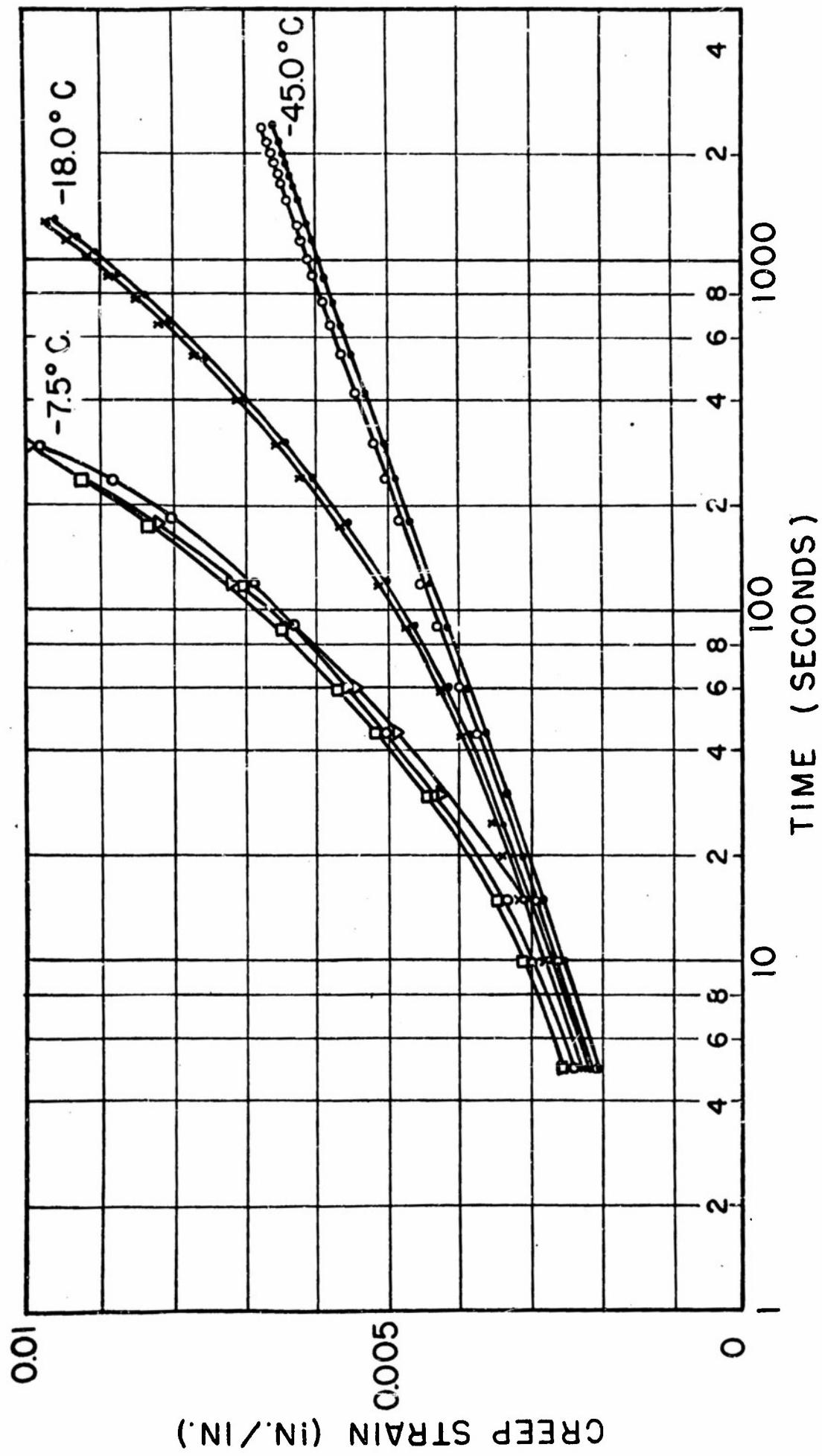


FIG. 5 CREEP CURVES OF A Zn SINGLE CRYSTAL UNDER A CONSTANT SHEAR STRESS OF 34.1 PSI AT THREE DIFFERENT TEMPERATURES PLOTTED IN A SEMI-LOG SCALE. (SAME DATA AS IN FIG. 4)

under constant stress, yet the transition time (i.e., the time when the curve begins to curve upward) was a function of temperature. For a better understanding of the phenomenon, a single crystal specimen was tested at three different temperatures under six different stresses. Fig. 6 to Fig. 11 show the results plotted on a semi-log scale. It seems apparent that the transition time varied with temperature in a regular manner under constant stress, but not with stress at constant temperature as tabulated below:

Temperature °C	Transition Time (in seconds) under Constant Stress (psi) of					
	24.9	27.6	30.7	33.1	36.2	39.6
0	60					
-10	240	30				
-18.6						70
-20	900	135	60	110	70	
-24						180
-31		720	350	600	360	
-35.3						1280
-39.6			1500	3000	1800	

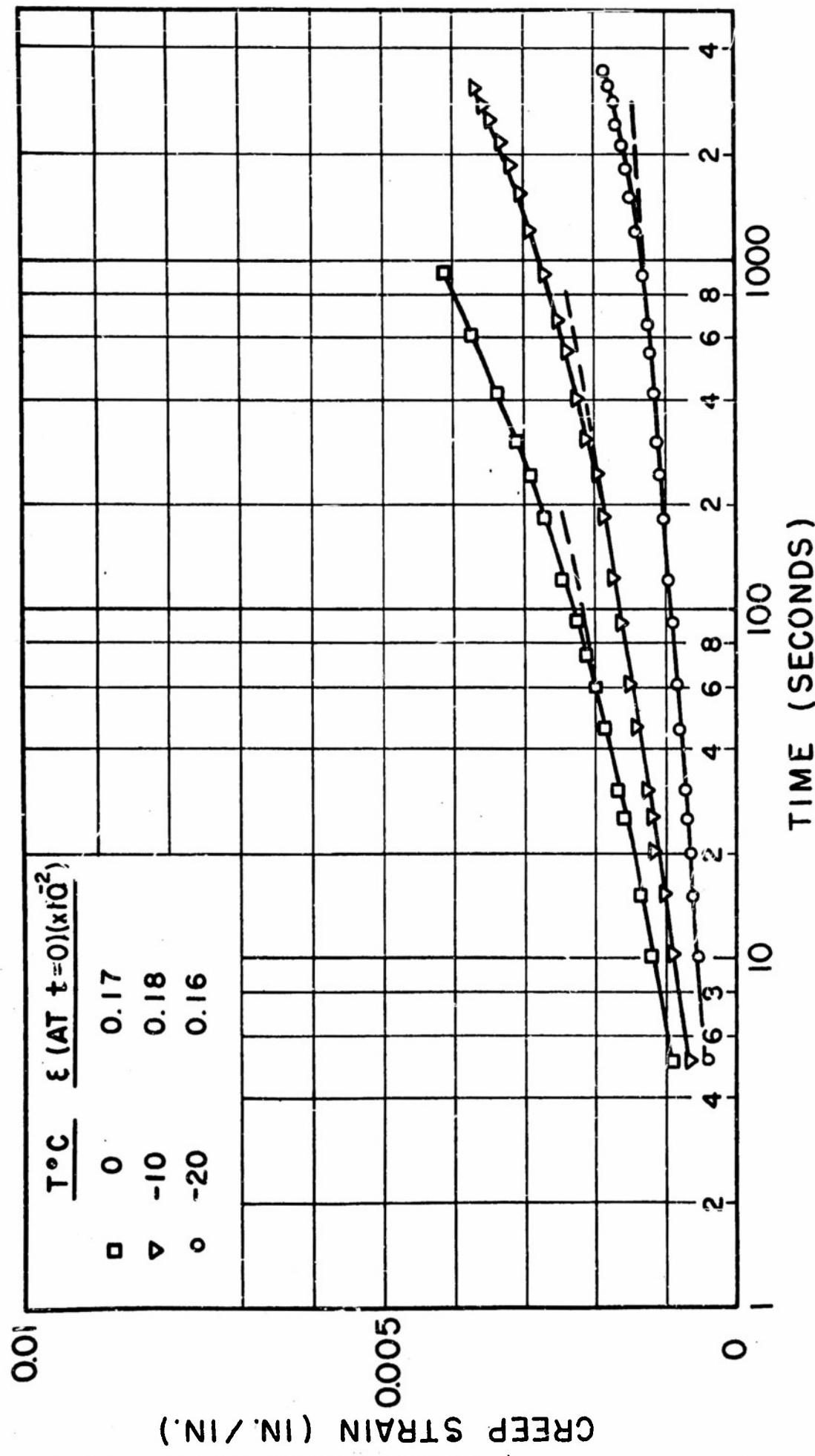


FIG. 6 CREEP CURVES OF A Zn SINGLE CRYSTAL UNDER A CONSTANT SHEAR STRESS OF 24.9 PSI AT THREE DIFFERENT TEMPERATURES.

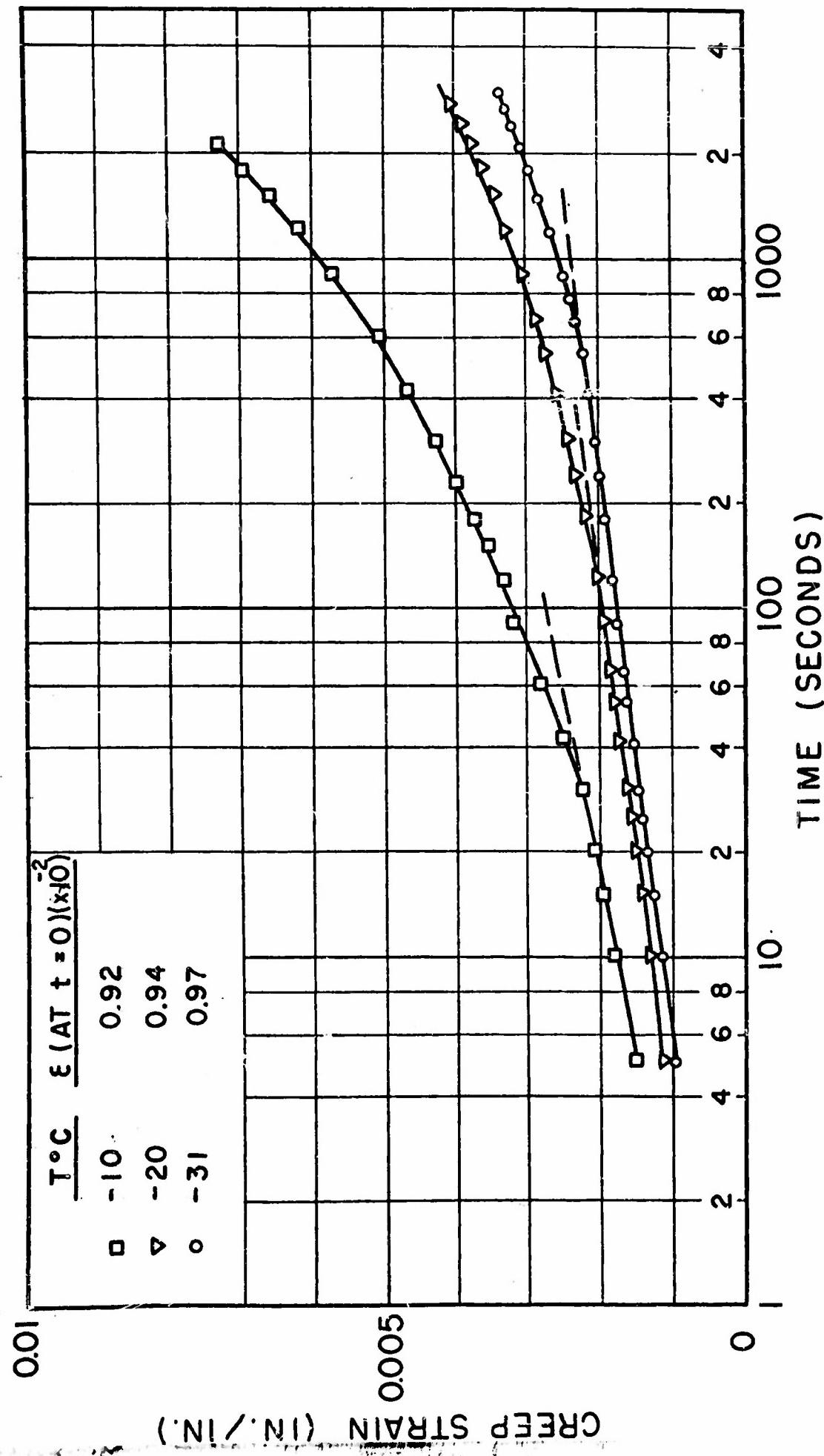


FIG. 7 CREEP CURVES OF A Zn SINGLE CRYSTAL UNDER A CONSTANT SHEAR STRESS OF 27.6 PSI AT THREE DIFFERENT TEMPERATURES.

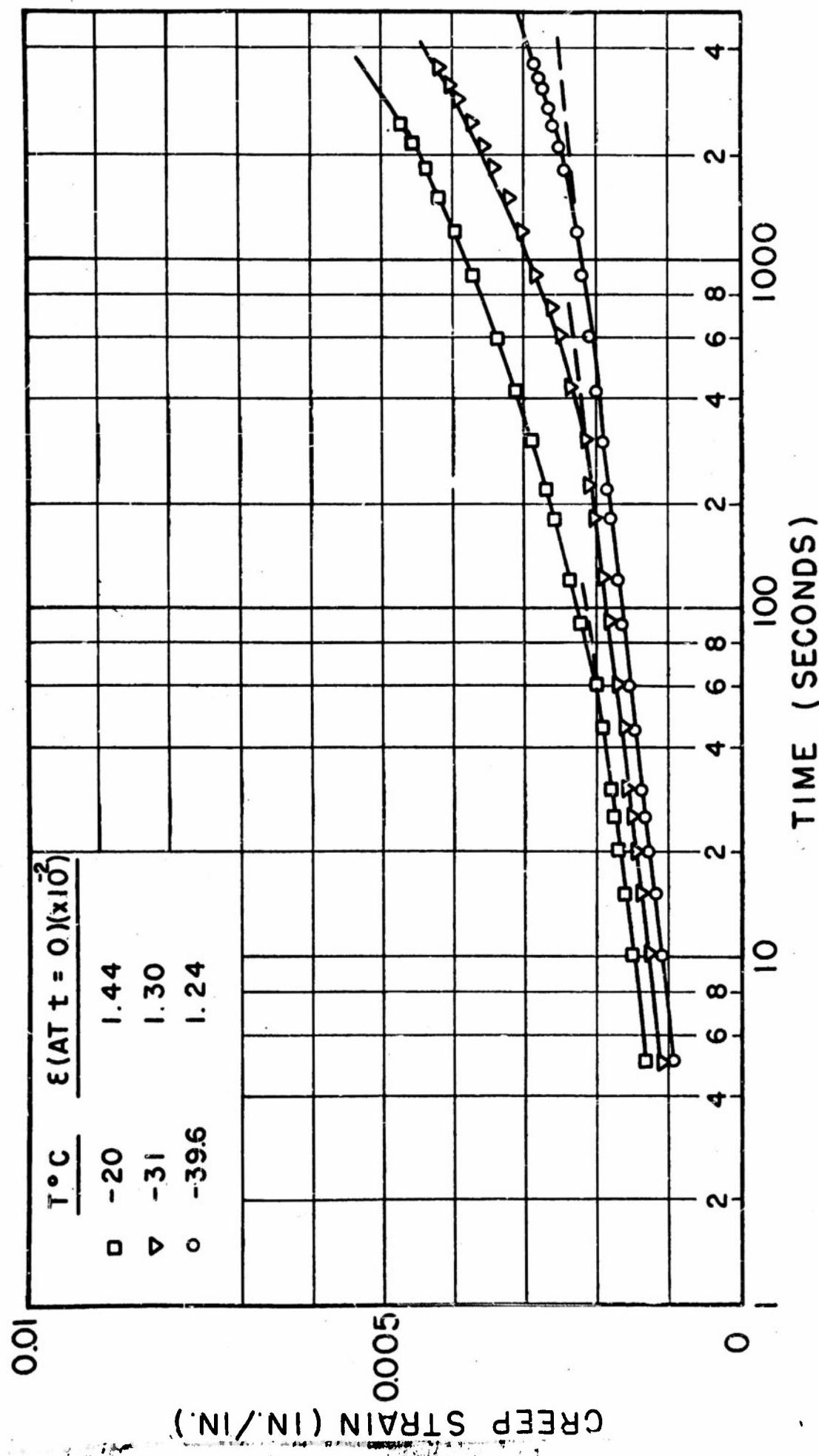


FIG. 8 CREEP CURVES OF A Zn SINGLE CRYSTAL UNDER A CONSTANT SHEAR STRESS OF 30.7 PSI AT THREE DIFFERENT TEMPERATURES.

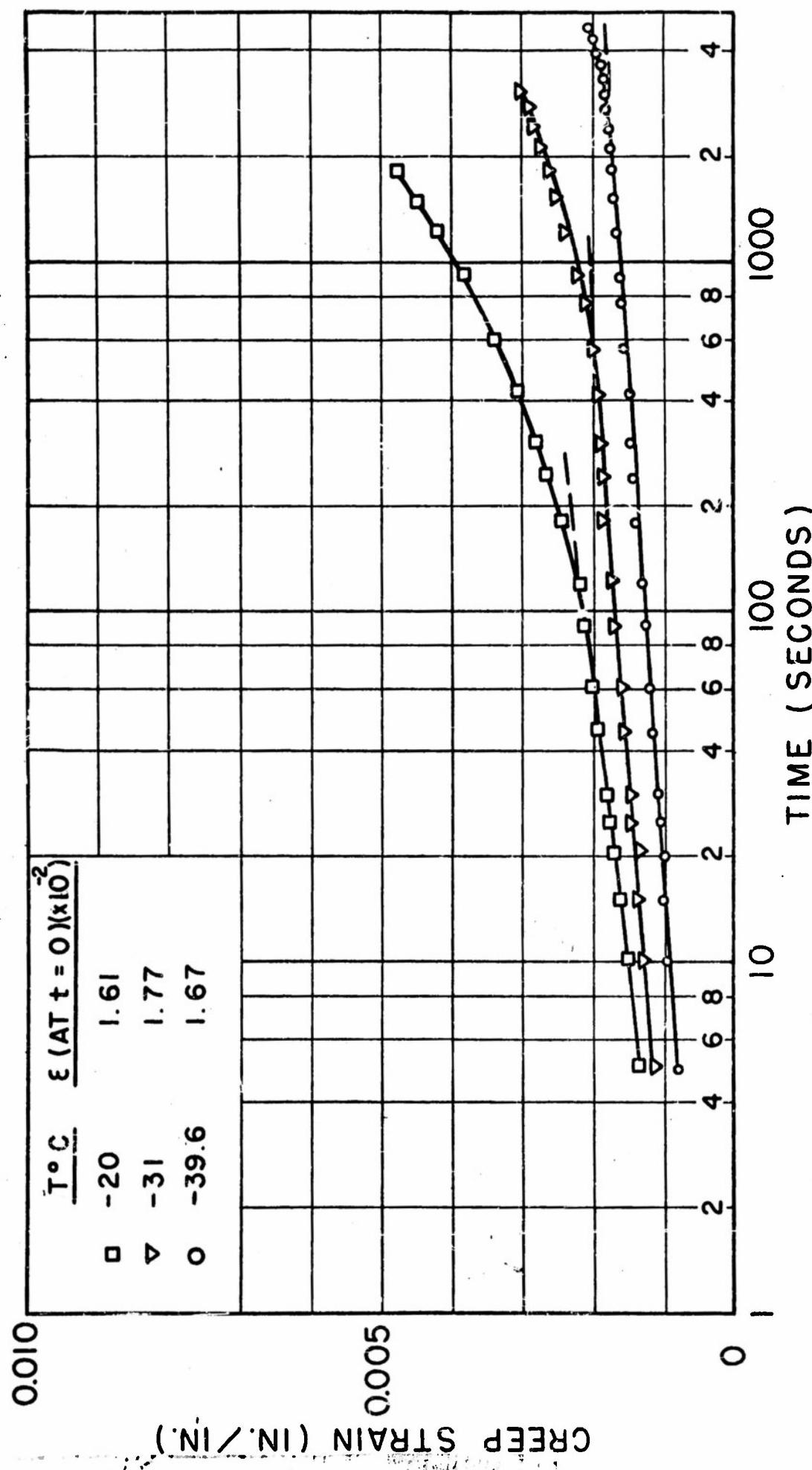


FIG. 9 CREEP CURVES OF A Zn SINGLE CRYSTAL UNDER A CONSTANT SHEAR STRESS OF 33.1 PSI AT THREE DIFFERENT TEMPERATURES.

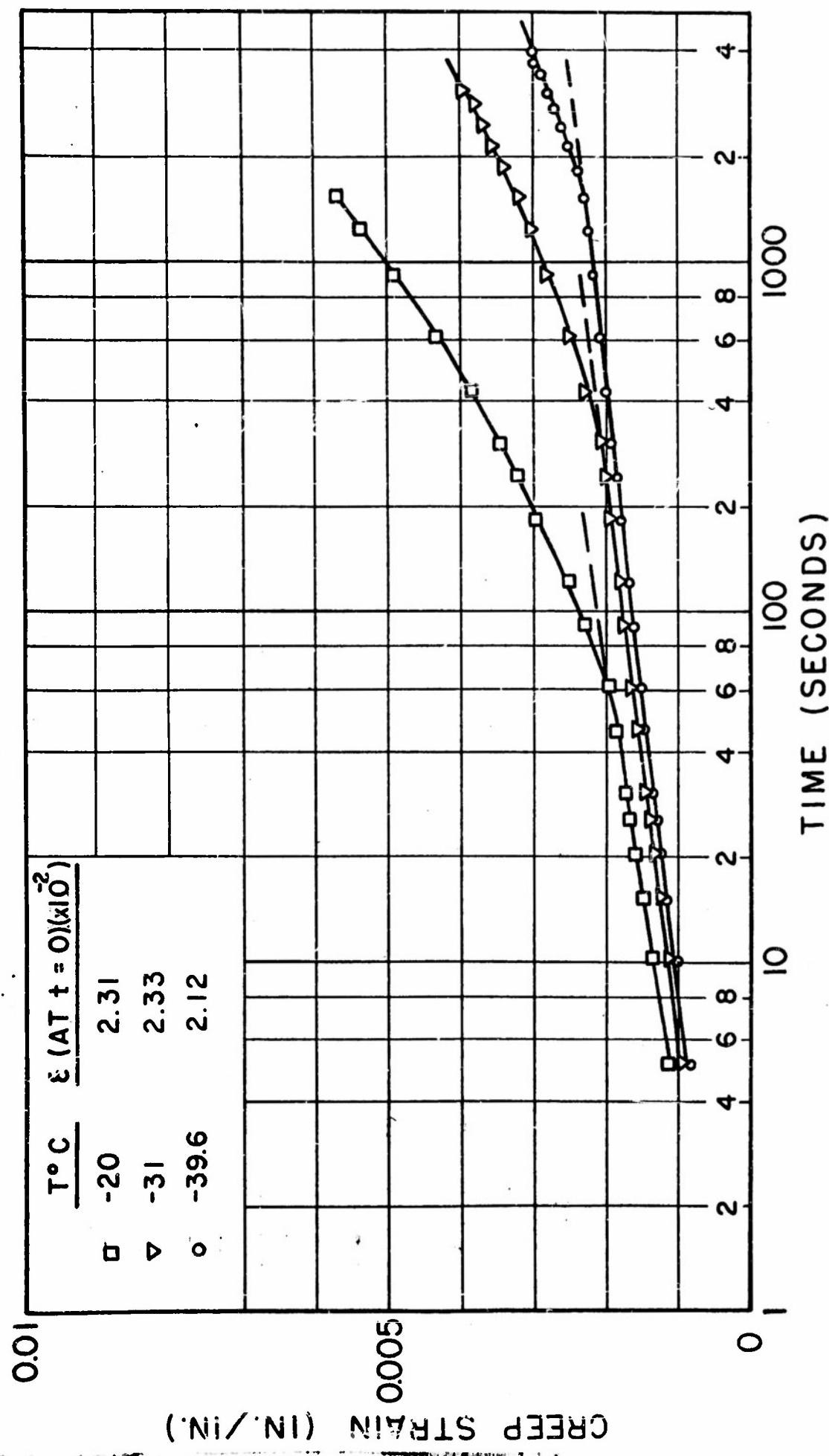


FIG. 10 CREEP CURVES OF A Zn SINGLE CRYSTAL UNDER A CONSTANT SHEAR STRESS OF 36.2 PSI AT THREE DIFFERENT TEMPERATURE

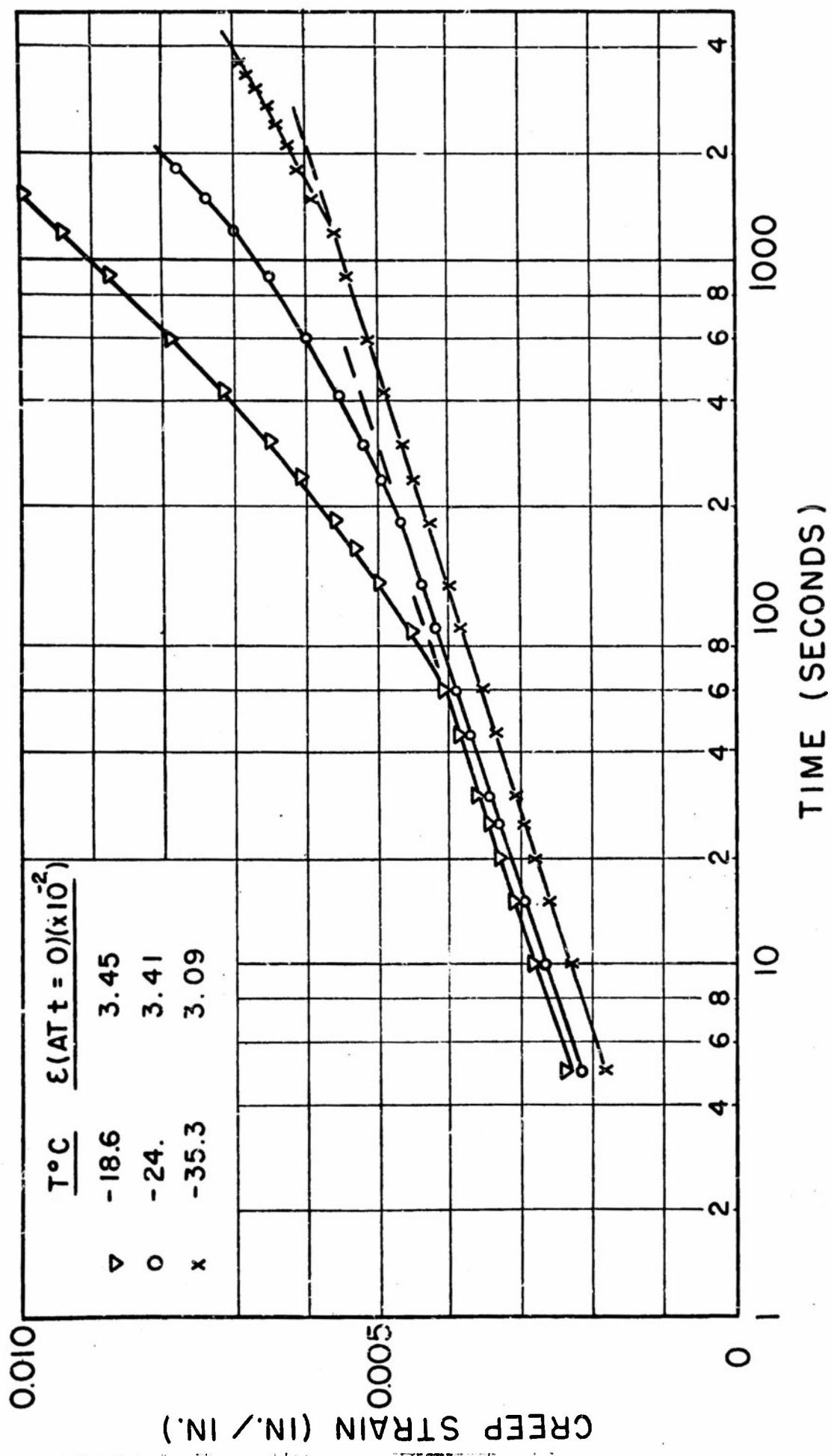


FIG. II CREEP CURVES OF A Zn SINGLE CRYSTAL UNDER A CONSTANT SHEAR STRESS OF 39.0 PSI AT THREE DIFFERENT TEMPERATURES.

### Discussion

At low temperatures the major contribution of creep strain occurs within the first few minutes. This strain, according to the exhaustion theory (11), must be due to the fast moving dislocations. The rate of motion of these dislocations is controlled by a thermal activation energy,  $Q$ . In its simplest form, as suggested by Mott (23), it is expressed as

$$Q = \frac{\gamma \epsilon}{\sigma_0}$$

where  $\gamma$  is a function of applied stress,  $\sigma_0$  is the stress at which movement of dislocations is possible without the help of thermal vibration, and  $\epsilon$  is the creep strain. Thus  $Q$  would be directly proportional to  $\epsilon$ . Hence, the creep strain during the first few minutes constitutes the major portion of the creep strain.

The results of the present investigation, however, point to an explanation of this phenomenon which seems to contradict the role of the thermal activation energy pictured above. According to the present results, the transition time can best be explained as governed by a diffusion process which controls the rate of climb of dislocations out of the slip planes (24). Fig. 12 is a plot of  $\log \frac{1}{t}$  against  $\frac{1}{T}$ , using the experimental results in the table shown above. The linear relationship between  $\log \frac{1}{t}$  and  $\frac{1}{T}$  is well established. Thus the following expression holds:

$$t_1 e^{-\frac{Q}{RT_1}} = t_2 e^{-\frac{Q}{RT_2}}$$

where  $t_1$  is the transition time at  $T_1$ , and  $t_2$  is the transition time at  $T_2$ .

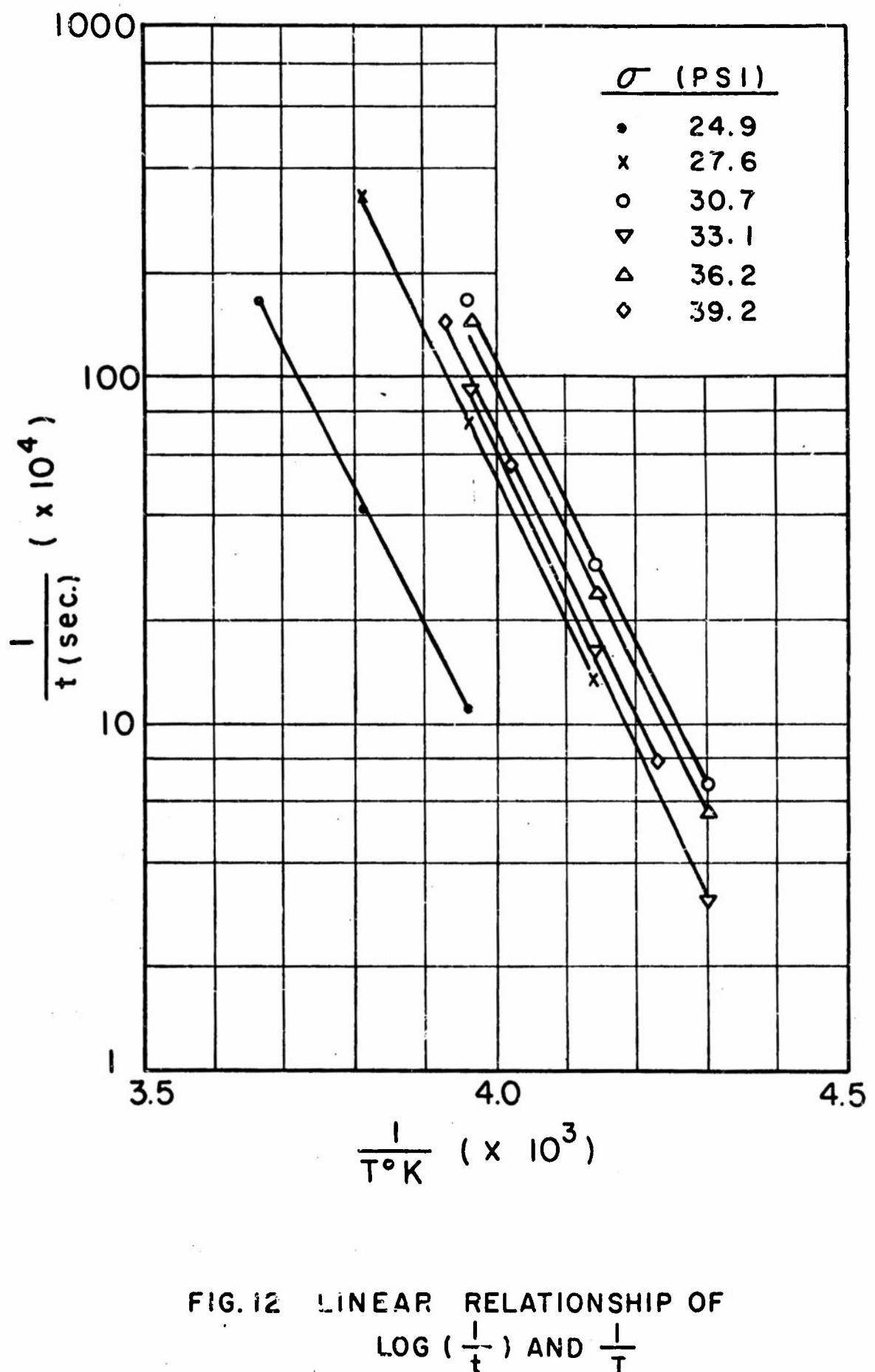


FIG. 12 LINEAR RELATIONSHIP OF  
 $\log(\frac{1}{t})$  AND  $\frac{1}{T}$

This formula is applicable because in the present investigation the experimental conditions were confined to such a narrow temperature range that the state of the material at different temperatures would be substantially the same at the transition time. Because the differences among the instantaneous strains under constant stress at different temperatures was within the range of scatter at the same testing temperature, it could be assumed that the total number of active Frank-Read sources was approximately the same, regardless of temperature. Also, because all the linear portions of the semi-log plots under constant stress were approximately parallel, it could be assumed that the nature of the lattice imperfections was about the same. It should be further noted that all the straight lines in Fig. 12, though characterized by different stresses, are parallel in spite of the fact. Hence, it seems apparent that the slopes are independent of stress. Furthermore, the activation energy calculated for the transition time was approximately 19,000 cal/mole, which is very close to the Q values obtained from self-diffusion along the C-axis (21,800 cal/mole<sup>(25)</sup>), from high temperature creep (20,000 cal/mole<sup>(26)</sup>), and from stress-induced motion of small angle boundary (21,500 cal/mole<sup>(5)</sup>).

In view of the foregoing, it would seem more appropriate to account for the phenomenon of decreasing rate of low temperature creep by development of a back stress, rather than by exhaustion of dislocations. It seems a reasonable assumption that by the time creep begins, a certain number of Frank-Read sources have already become active in generating dislocations. The fact that the rate of generating new dislocations decreased is more probably due to the back stress produced by the piling up of former dislocations on the lattice imperfections. Edwards and Washburn<sup>(17)</sup> have shown

that at liquid nitrogen temperature back stress increases with plastic strain. It should be emphasized here that creep tests were performed under constant stress, which means that the driving force for generating dislocations remained constant, whereas the back stress increased with time. As the back stress increased, the creep rate decreased. The role that thermal energy plays in this process seems to be that it decreases the back stress by releasing the piled-up dislocations. This seems to occur by "dislocation climbing", a process which holds particularly true for high temperature creep. The significance of the transition time as a function of temperature lends support to this argument.

### Summary

Reproducibility of the creep curves makes it possible to discuss creep with more assurance. The necessary conditions for reproducibility seem to be: (1) annealing at a temperature close enough to the melting point, and (2) a consistent loading time involving negligible impact for the creep tests.

The transition time, defined as the time at which the creep curve deviated from the logarithmic creep, was found to be temperature-dependent. The creep at the transition time was governed by diffusion. The calculated activation energy was 19,000 cal/mole, and independent of the applied stress.

For the phenomenon of low temperature creep back stress seems to offer a more probable explanation than the exhaustion of dislocations.

### Acknowledgment

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